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(दूसरा पुनरीक्षण)

Indian Standard

DIPENTENE FOR PAINTS — SPECIFICATION

(Second Revision)

UDC 667·629·2 : 66·062·21·596·7

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 4

Raw Materials for Paint Industry Sectional Committee, CHD 021

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard was first published in 1955 based on data available for imported Dipentene. However, the indigenous production of dipentene commenced in the early 1960's. Consequently, there is practically no import of dipentene since 1960. This standard was therefore revised in 1969 in order to take care of indigenous technology. In the first revision, requirements for distillation range and flash point had been modified. Additional requirements for residue on evaporation, copper corrosion, unpolymerizable matter, grease spot test and kauri-butanol value were included.

The indigenous Dipentene is a by-product of the manufacture of Camphor from Alpha-Pinene. With the advancement of technology of production and on the basis of experience gained, the committee now felt to revise this standard. In this second revision description, requirements for flash point, residue on evaporation, refractive index, relative density, distillation range and unpolymerizable matter have been modified to suit the currently available material. The temperature of reporting for relative density and refractive index has been changed from 30°C to 27°C. A new requirement of ash content has been added and requirement for neutrality has been changed to acid value.

A study has been taken up to find out the alternate for Kauri reagent to enable testing of Kauri Butanol value as given in the requirements of this standard. It has become necessary because of difficulty in availability of Kauri resins.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
DIPENTENE FOR PAINTS — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for dipentene used as a thinner and solvent for paints.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
82 : 1973	Method of sampling and test for thinners and solvent for paints (*first revision*)
266 : 1977	Sulphuric acid (*second revision*)
354 (Part 1) : 1987	Methods of sampling and test for resins for paints : Part 1 General test method (*second revision*)
361 : 1973	Normal butyl alcohol, technical (*second revision*)
537 : 1967	Toluene, pure, nitration grade (*first revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1303 : 1983	Glossary of terms relating to paint (*second revision*)
1997 : 1982	Specification for burettes (*second revision*)
4905 : 1968	Methods for random sampling

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given under **2** of IS 82 : 1973, IS 1303 : 1983 and the following shall apply.

3.1.1 *Kauri-Butanol Value*

The volume in millilitres at 25°C of the solvent, corrected to a defined standard, required to produce a defined degree of turbidity when added to 20 g of a standard solution of Kauri resin in normal butyl alcohol. For Kauri-butanol value of 60 and above the standard is toluene and has an assigned value of 105. For Kauri-butanol values below 60, the standard is a blend of 75 percent n-heptane and 25 percent toluene and has an assigned value of 40.

4 REQUIREMENTS

4.1 Description

4.1.1 The material shall be a mixture of monocyclic terpenes, namely, Alpha terpinene, Limonene, Gamma terpinene and Terpinolene; bicyclic terpenes, namely, Car-3-ene and Para-cymene with characteristic odour. It shall be clear and free from sediment, suspended matter and undissolved water.

4.1.2 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable containers as agreed to between the purchaser and the manufacturer.

5.2 Marking

5.2.1 The containers shall be marked with the following:

a) Name of the material;

b) Indication of the source of manufacturer;

c) Net volume;

d) Batch number; and

e) Month and year of manufacture.

6 Sampling

6.1 The method of drawing representative samples of dipentene for paints, shall be as prescribed in Annex G.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Dipentene for Paints

(*Clause* 4.1.2)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	Clause No. in Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Colour	Not darker than a freshly prepared solution of 0.003 of Potassium Dichromate in one litre of distilled water	—	5 of IS 82 : 1973
ii)	Grease spot test	To pass the test	A	—
iii)	Flash point, *Min*	35°C	—	IS 354 (Part 1) : 1987
iv)	Ash, percent by mass, *Max*	0.01	B	—
v)	Residue on evaporation, percent by mass, *Max*	2.00	—	8 of IS 82 : 1973
vi)	Refractive index* at 27°C	1.470 0 to 1.480 0	C	—
vii)	Relative density,† 27°/27°C	0.843 0 to 0.855 0	—	6 of IS 82 : 1973
viii)	Distillation range, Initial b.p. For 95 ml	*Min* 170°C *Max* 195°C	—	7 of IS 82 : 1973
ix)	Unpolymerizable matter, percent by volume, *Max*	15	D	—
x)	Acid value, *Max*	1.0	E	—
xi)	Corrosive sulphur (Copper corrosion)	Shall not blacken or corrode clean copper strip	—	11 of IS 82 : 1973
xii)	Kauri-butanol value, *Min*	60	F	—

*The refractive index determined at any temperature within the range 20° to 30°C can be adjusted to 27°C using the correction factor of ±0.000 46 for every degree celsius fall and rise of temperature.

†The relative density is synonymous with specific gravity. It may be determined at any temperature within the range of 20° to 30°C and can be adjusted to 27°/27°C using the correction factor of 0.000 8 for every degree celsius fall and rise of temperature.

ANNEX A

[*Table* 1, *Item* (ii)]

GREASE SPOT TEST

A-0 GENERAL

A-0.1 Outline of the Method

A few drops of the material is evaporated on a filter paper and observed for any oily spot on it.

A-1 Apparatus

A-1.1 Porcelain Basin

Evaporating, with spout, flat bottom, 70 mm in diameter [*see* IS 2837 (Part 2) : 1977].

A-2 PROCEDURE

A-2.1 Place 5 drops of the material by means of a small pipette at the centre of a clean white filter paper supported on the porcelain basin and allow the liquid to evaporate at room temperature, away from direct sunlight. Examine after 30 minutes whether there is any oily or greasy spot on the filter paper.

A-3 REPORT

A-3.1 The material shall be deemed to have passed the test if no oily or greasy spot is seen on the filter paper.

ANNEX B

[*Table* 1, *Item* (iv)]

DETERMINATION OF ASH

B-0 GENERAL

B-0.1 Outline of the Method

Ash content is determined by igniting the material, under specified conditions and weighing the non-combustible material.

B-1 APPARATUS

B-1.1 Porcelain Crucible or Dish

B-1.2 Muffle Furnace — Range up to 1 200°C.

B-2 PROCEDURE

B-2.1 Weigh a clean and previously ignited porcelain crucible or dish, accurately to the nearest 0.000 1 g. Add 10 to 25 ml of the material and weigh again. Heat the material gently by bringing a flame on the bottom and sides of the crucible until the oil ignites. Reduce the size of the flame until the heat is just sufficient to keep the material burning. Continue heating of the sample to a black char and transfer it to a muffle furnace or heat it over a burner until all carbonaceous matter is reduced to ash. Remove the crucible, place in a desiccator and cool to room temperature. Weigh the repeat heating and cooling until the difference between two weighings is less than 1 mg.

B-3 RESULTS

B-3.1 The material shall be declared to have passed the test if the ash content does not exceed 0.01 percent by mass.

ANNEX C

[*Table* 1, *Item* (vi)]

DETERMINATION OF REFRACTIVE INDEX

C-0 GENERAL

C-0.1 Outline of the Method

Refractive index of the material is determined with an Abbe's refractometer.

C-1 APPARATUS

C-1.1 Refractometer

Abbe's refractometer. The temperature of the refractometer shall be controlled within ± 0.1°C and for this purpose, it shall be provided with a thermostatically controlled water-bath and a motor-driven pump to circulate water through the instrument. The instrument shall be standardized, following the manufacturer's instructions with a liquid of known purity and refractive index or with a glass prism of known refractive index. Distilled water, which has a refractive index of 1.333 0 at 20.0°C, is a satisfactory liquid for standardization.

C-1.2 Light Source

If the refractometer is equipped with a compensator, a tungsten lamp or a daylight bulb shall be used. Otherwise, a monochromatic light, such as an electric sodium vapour lamp, shall be used.

C-2 PROCEDURE

C-2.1 Filter the material through a filter paper to remove any impurities and the last traces of moisture. Make sure that the sample is completely dry. Adjust the temperature of the refractometer to 30.0° ± 1°C. Ensure that the prisms are clean and completely dry, and then place a few drops of the material on the lower prism. Close the prisms, tighten firmly with the screw-head and allow to stand for one or two minutes. Adjust the instrument and light to obtain the most distinct reading possible and determine the refractive index.

ANNEX D

[*Table* 1, *Item* (ix)]

DETERMINATION OF UNPOLYMERIZABLE MATTER

D-0 GENERAL

D-0.1 Outline of the Method

The amount of unpolymerizable matter left after action of sulphuric acid of specified concentration on the material is determined.

D-1 APPARATUS

D-1.1 Measuring Cylinder

Stoppered, of 50 ml capacity (*see* IS 878 : 1975).

D-2 REAGENT

D-2.1 Concentrated Sulphuric Acid

98 percent (*w/w*) (*see* IS 266 : 1977).

D-3 PROCEDURE

D-3.1 Add exactly 10 ml of the material in drops to 20 ml of sulphuric acid contained in the 50-ml cylinder immersed in ice water and keep the cylinder in ice water for about 15 minutes. Shake the cylinder vigorously during addition of the material. Remove the cylinder from ice water, shake well, immerse in a water-bath and heat at 60° to 65°C for about 10 minutes with frequent shaking of the contents. Return the cylinder to cold water-bath to attain room temperature. Add sufficient amount of sulphuric acid to bring the level of the liquid of the upper layer into the middle and calculate the unpolymerized matter as percentage by volume of the original sample.

ANNEX E

[*Table* 1, *Item* (x)]

DETERMINATION OF ACID VALUE

E-1 APPARATUS

E-1.1 Conical Flask

100 to 250 ml capacity.

E-1.2 Measuring Cylinder

25 ml capacity.

E-1.3 Burette

25 ml capacity graduated in lengths of a millilitre (*see* IS 1997 : 1982).

E-2 REAGENTS

E-2.1 Ethyl Alcohol

95 percent by volume, or rectified spirit (*see* IS 323 : 1959). To remove impurities which might get coloured by potassium hydroxide solution and interfere with the sharpness of the end point, reflux the 95 percent alcohol or rectified spirit over solid sodium hydroxide for 6 to 8 hours, and then distil.

E-2.2 Potassium Hydroxide Solution

0.1 N approximately.

E-2.3 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

E-2.4 Phenol Red

Dissolve 0.1 g of phenol red in 100 ml of rectified spirit.

E-3 PROCEDURE

E-3.1 Weigh about 2.5 g of dried material and dissolve in 20 ml of rectified spirit (**E-1.1**) previously neutralized to phenolphthalein indicator with potassium hydroxide solution. Add about 10 drops of indicator except in the case of phenolic perfumery material and titrate with potassium hydroxide solution until the solution remains fantly pink after 10 seconds of shaking. The flask and its content may be retained for the determination of ester value. If the perfumery material under examination contains phenols or compound with phenolic groups, use phenol red as indicator instead of phenolphthalein.

E-3.2 Calculation

$$\text{Acid value} = \frac{56.1 \times V \times N}{M}$$

where

V = volume, in ml, of standard potassium hydroxide solution;
N = normality of the potassium hydroxide solution; and
M = mass, in g, of the material taken for the test.

NOTES

1 Sometimes, certain samples of perfumery materials, such as oils of vetiver and geranium, have very high acid values (20 and above). In such case 2.5 g of the material requires even more than 10 ml of 0.1 N aqueous potassium hydroxide solution. With highly acidic materials, it is recommended that 0.75 to 1 g of the material be taken instead of 2.5 g.

2 In case of benzyl formate which may get hydrolyzed under the above condition, it is desirable to cool the sample below 5°C before titration.

ANNEX F

[*Table* 1, *Item* (xii)]

DETERMINATION OF KAURI-BUTANOL VALUE

F-0 GENERAL

F-0.1 Outline of the Method

This test is intended for determination of relative solvent power of the material. The material is titrated against a standard kauri-butanol solution to an end point where the sharp outlines of 10 point print placed below the water-bath become blurred. From the volume of the material consumed, the kauri-butanol value is calculated.

F-1 APPARATUS

F-1.1 Water Bath

Clear glass vessel.

F-1.2 Flasks

One 200-ml capacity volumetric flask and one 250-ml capacity long-necked flask.

F-1.3 Burette

50 ml capacity.

F-1.4 Print Specimen

A sheet of white paper having on it black 10 or 12 point print, monotype Baskerville; a specimen of the print is given below:

> The volume in millilitres at 25°C of the solvent, corrected to a defined standard, required to produce a defined degree of turbidity when added to 20 g of standard solution of kauri resin in n-butyl alcohol. For kauri-butanol values of 60 and above, the standard is toluene and has an assigned value of 105. For kauri-butanol values below 60, the standard is a blend of 75 percent n-heptane and 25 percent toluene and has an assigned value of 40.

F-2 REAGENTS

F-2.1 Kauri-Butanol Solution

Place in a 3-litre flask 400 g of clean, pale, bold kauri resin ground to pea-size. Add, white shaking vigorously, 2 000 g of n-butyl alochol conforming to IS 361 : 1973. Reflux the contents over a steam-bath till the resin goes into solution. Allow the solution to stand for 48 hours and then filter through a Buchner funnel using double filter paper.

F-2.2 Toluene

Conforming to IS 537 : 1967.

F-2.3 Heptane-Toluene Blend

Consisting of 25 ± 0.1 percent toluene and 75 ± 0.1 percent n-heptane.

F-3 PROCEDURE

F-3.1 Standardization of Kauri-Butanol Solution

Weigh accurately 20 ± 0.10 of the kauri-butanol solution in the 250-ml long necked flask and keep it in the water-bath maintained at 25°C. Titrate the contents with standard toluene with constant swirling keeping the temperature at 25°C. Continue the titration with successive reduced amount of toluene as the end point is approached. The end point is reached when the sharp outlines of 10 point print placed below the water-bath and observed through the liquid are obscured or blurred. Alternately, if the titration is done in a room whose temperature is controlled at 25° ± 1°C, the 10 point print is placed below the flask itself and end point is viewed through the contents of the flask. Check the temperature of the bath immediately and repeat the experiment if the variation is more than 1°C from 25°C. Note the volume of toluene used and this represents the actual titer for the particular kauri-butanol solution.

NOTE — This value should preferably lie between 100 to 110 ml. Adjust the concentration of the solution by dilution if these limits are exceeded.

F-3.2 Standardization of Heptane-Toluene Blend

Weigh accurately 20 ± 0.10 g of the kauri-butanol solution (**F-2.1**) in a 250-ml long-necked flask, keep in the water-bath maintained at 25°C and titrate against the heptane-toluene blend. Alternatively, the titration may be carried out in a room whose temperature is controlled at 25°C ± 1°C. Note the volume of heptane-toluene blend required to give a blurred vision of the 10 point print when viewed through the liquid.

NOTE — If the composition of the blend is known to differ from 25 ± 0.1 percent toluene but is within the range 22 to 28 percent toluene, the constant in the blend factor equation (**F-4**) will differ from 40.0 by 0.60 units for each one percent toluene. For example, at 28 percent toluene, the constant is 41.8.

F-3.3 Weigh accurately 20 ± 0.10 g of the kauri-butanol solution (**F-2.1**) a 250 ml long-necked flask. Place the flask on a water-bath maintained 25° ± 1°C. Fill the 50-ml burette with the material and titrate this against the kauri-butanol solution with constant stirring. The end point reached when the sharp outlines of 10 point print placed directly below the water-bath or the flask, in case the titration is done in a room whose temperature is controlled at 25°C ± 1°C, and observed through the liquid are blurred. Check the temperature immediately and repeat the experiment if the variation is more than 1°C. Note the volume of the material consumed.

F-4 CALCULATION

$$\text{kauri-butanol value} = \frac{65\,(C - B)}{A - B} + 40$$

where

C = volume, in ml, of the material (dipentene) required to titrate 20 g of kauri-butanol solution (**F-3.3**);

B = volume, in ml, of heptane-toluene blend required to titrate 20 g of kauri-butanol solution (**F-3.2**); and

A = volume, in ml, of toluene required to titrate 20 g of kauri-butanol solution (**F-3.1**).

NOTE — If the burette is maintained at a temperature other than 25°C, correct the volume of the titrant used to standard temperature as follows:

$$\text{Correction, per ml} = v\,(25 - I) \times 0.000\,9$$

where

v = volume, in ml, of titrant used; and

I = temperature of titrant in the burette in °C.

ANNEX G

(*Clause* 6.1)

SAMPLING OF DIPENTENE FOR PAINTS

G-1 SAMPLING

G-1.1 General Requirements

While drawing samples, the following precautions and directions should be adhered to.

G-1.1.1 Samples shall not be taken in an exposed place.

G-1.1.2 The sampling apparatus shall be clean and dry when used.

G-1.1.3 The samples shall be placed in clean, dry and airtight containers made of suitable material on which dipentene has no action.

G-1.1.4 Once the container is opened, there is a risk of deterioration of contents due to the presence of air. Care should be taken to avoid risk of polymerization of the sample by undue exposure to air or strong light.

G-1.1.5 After filling, the sample containers shall be marked with all details of sampling.

G-2 SCALE OF SAMPLING

G-2.1 Lot

All the containers of the same capacity containing dipentene produced under similar condition of manufacture shall constitute a lot. If the material is supplied in bulk in drums of capacity 500 litres or more, each drum shall constitute a lot.

G-2.1.1 For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

G-2.2 In the case of containers of capacity less than 500 litres, the number of containers selected from each lot shall depend on the size of the lot and shall be in accordance with Table 2.

G-2.3 These containers shall be selected at random from the lot as per IS 4905 : 1968.

Table 2 Scale of Sampling
(*Clause* G-2.2)

Lot Size N (1)	Number of Containers to be Selected n (2)
Up to 25	2
26 to 50	3
51 to 100	5
101 to 300	8
301 and above	13

G-3 PREPARATION OF TEST SAMPLES

G-3.1 From each of the selected containers a representative portion of the material shall be taken as per **5.3.2** and **5.3.3**, which shall be sufficient for carrying out tests for all the characteristics given in the specification.

G-3.2 Individual Sample

G-3.2.1 A sample of 200 ml of the liquid shall be withdrawn from each selected containers as per **5.3.1** and kept in separate sample containers of such size that they are nearly filled by the sample. These are termed as individual samples.

G-3.2.2 When the sample is supplied in drums of capacity 500 litres or more, samples shall be collected from each drum at five different depths with the help of a suitable sampling instrument. These five samples of 200 ml each, shall be kept in separate sample containers of such size that they are nearly filled by the sample.

G-3.3 Composite Sample

Out of the portions (*see* **G-3.2**), a small but equal quantity of the material shall be taken as mixed thoroughly to form a composite sample. In the case of material supplied in drums, the composite sample shall be prepared by mixing thoroughly the portions collected at different depths.

G-4 NUMBER OF TESTS

G-4.1 Each of the individual samples shall be examined for the colour of the solvent and Kauri-butanol value. The test for all other characteristics shall be conducted on composite sample.

G-5 CRITERIA FOR CONFORMITY

G-5.1 The lot shall be deemed to conform to the specification if all the test results according to **G-4** satisfy the corresponding requirement given in Table 1.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, *1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.